RESEARCH ARTICLE

Surface Modified Calotropis Gigantea Fiber Reinforced Polypropylene Composites

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ABSTRACT: In this article, the effects of chemical treatment (sodium dodecyl sulfate, SDS, and SDS pretreated benzoyl chloride) on the physicomechanical, morphological, and weather tests of calotropis gigantea fiber (CGF) reinforced polypropylene (PP) composites were investigated. The outcomes show that CGFs act as reinforcing fillers enhancing the physicomechanical properties (e.g. tensile strength, tensile modulus, and impact strength, hardness, and water absorption (WA)) of composites. PP/CGF composites with benzoyl chloride after SDS pretreatment indicate better mechanical properties than SDS treated and untreated fiber composites. The interfacial properties were examined by scanning electron microscopy (SEM) and it was performed that the interfacial interaction between PP and CGF was enhanced due to the treatment of fibers, which verified the obtained mechanical properties of the composites. The water absorption test showed that SDS pretreated benzoyl chloride-treated CGF composites exhibited less water absorption than both SDS-treated and untreated composites. Weather tests have shown that SDS pretreated benzoyl chloride-treated composites exhibited less loss of tensile strength and tensile modulus compared to both SDS-treated and untreated composites in terms of their decay time.

KEYWORDS: Composite, calotropis gigantea fiber, polypropylene, SDS-treatment, benzoyl chloride treatment

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1. INTRODUCTION

A significant part of this scientific research on new materials has concentrated on the usage of natural fibers (NFs) to strengthen composite materials instead of synthetic fibers (SFs) [1, 2]. Researchers are currently focusing on the use of cellulose fibers (CFs) and agro-residues for strengthening in various thermoplastic (TP) and thermoset (TS) resins considering high strength, high hardness, and low concentration of weight ratio [3, 4]. Due to the great and outstanding combination of physical and mechanical features, natural fiber composites are now widely used in a wide variety of fields and are used in civil construction, machinery construction, electrical and electronic apparatus, automobile industrial factory, in making aircraft, and much more [5-8]. Much research has been done on synthetic composites of synthetic matrix-based synthetic fibers (e.g., glass, carbon, nylon, and Kevlar) [9, 10]. SF reinforced TP or TS composites are effective on NF strengthened composites due to their advanced strength, durability, deterioration, and moisture resistance features. Scientists prefer TP composites to TSs due to lower production costs and lower processing. The use of SFs is a great environmental problem because these fibers are not sterile. Due to increasing environmental awareness, the matrix is being explored day by day as lignocellulosic components are made from fibers and TP/TS polymers as composites.

NFs have some benefits over conventional plastic or synthetic components in terms of low price, low-density, suitable definite strength, renewability, recyclability, and biodegradability. Of all the NFs, we chose calotropis gigantea fiber (CGF) because it has sufficient buoyancy, hydrophobic-oleophilic features, high oil absorption capacity, and oil-water separation efficiency and is used as an oil-absorbing ingredient in the oil separation process to remove oil from water. CGF belongs to the family of *Asclepiadaceae* and lives in China, India, Bangladesh, and various portions of the

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world. This fiber has excellent biocompatibility and non-toxic properties [11,12]. The application of CGF is found mainly in the textile industry as a raw material with intrinsic biodegradability, and to date, very little research has been done on CGF-reinforced composites [13]. CGF plant grows up to 3-5 m in height and forms waxy flowers in clusters. Furthermore, it has oval-shaped pale green leaves and stalks with a milky nature, which is further enriched by fibers. The fibers obtained from the stems of the CG plant are used for composite production. Extract CG bark fiber contains 73.8% cellulose, 20.5% hemicellulose, 2.7% lignin, and 1.18% waxy substances [14]. When selecting new fibers, chemical elements performance a vital role in determining the mechanical features of the fiber in the NF composite. Table 1 lists the chemical components of NFs that are already used as reinforcements in NF composites with CG fibers.

Although cellulose fibers have multiple benefits, cellulose fibers reduce the adverse effects of high temperatures and the ubiquity of moisture absorption. Hydroxyl groups represent cellulose to form different hydrogen bonds and CFs to form hydrophilic. Manufactured composites, including nonpolar (hydrophobic) TP matrix and polar (hydrophilic) NFs, reduce mechanical features due to weaknesses in the plastic material and fiber [15]. The mechanical features of the composites can be progressed by modifying the NFs by chemical treatment [16]. The chemicals can interact effectively with cellulose and activate the hydroxyl group, which can induce the necessary features of the polymer. SDS is well-known as an anionic surfactant and is extensively used in the synthesis of biomaterials [17]. The presence of SDS in CF is expected to reduce the hydrophilic character as an outcome of chemical interactions between the polar main group of SDS and the polar group of fibers [18]. In contrast, benzoyl chloride is often used in fiber treatment. Benzoyl chloride contains benzoyl ($C_6H_5C=O$) which is applied with hydrophobic matrix to reduce the hydrophilic character of NF and improve the interaction with hydrophobic matrix [19].

In this study, PP was selected as a TP resin because it has several outstanding features such as transparency, superior surface strength, high impact strength, high heat distortion temperature, and dimensional stability. PP is moreover very fit for filling, strengthening, and mixing. NFs with PP are one of the possible roots for making natural or synthetic polymer composites. In this present study, unidirectional PP/CGF composites with different fiber contents were manufactured and their physicomechanical properties were evaluated. Both SDS treatment and the resulting SDS pretreated benzoylation treatment were used to improve adhesion between these two opposite surfaces. To date, no research work has reported on the physicomechanical properties of the composite of SDS-pretreated and subsequent benzoylation treated calotropis gigantea fiber (CGF). The interfacial properties were evaluated by analysis of SEM. Water absorption capacity and weather tests were also observed for optimized composites to investigate the suitability of composites for a wide-ranging of applications.

2. EXPERIMENTAL

Materials and Methods

Granules of homopolymer PP (trade name: Cosmoplene) were supplied by Polyolefin Company, Private Ltd., Singapore. SDS, benzoyl chloride and ethanol were obtained from Sigma-Aldrich Chemical Co. Inc. (Louis, USA). Calotropis gigantea plants were collected from local sources in Bangladesh. Trees older than one year seem to be more suitable for fiber extraction. The leaves along with the stems were removed and the stems were cut to the required length. It was then dried for three days at room temperature and then the steams were extracted by hand decorticated method. CG plants and extracted fibers are displayed in Figure 1. The properties of CG fibers were mentioned in Table 1.

Figure 1 Extraction of CGF: (a) CG plant, (b) removal of stem outer layer, and (c) extracted CGF

Table 1 Features of CG fiber

Description	Values
Physical properties	
Density (g/cc)	1.360
Diameter (μm)	90-260
Chemical compositions	
Cellulose (%)	73.8
Hemicellulose (%)	20.5
Lignin (%)	2.7
Wax (%)	1.18
Mechanical properties	
Tensile strength (N)	7.95
Percentage of elongation (%)	5.03

The SDS powder (5wt% of CGF) was dissolved in ethanol. Then, CGF was slowly added to the solution for 2h at room temperature. The modified fibers were filtered and dried in an oven at 70°C for 24h to remove the ethanol residue. One hundred fifty mL of benzoyl chloride was dissolved in 2L of 5% SDS solution and pretreated CGFs were immersed in the solution for 1 h. After modification, benzoylated fibers were filtered and soaked in ethanol for thirty min to remove unreacted reagents, and then dried in an oven at 80°C for about 24 h to constant weight. A set of formulations was arranged and the formulated compositions are presented in Table 2.

Preparation of CGF-Reinforced PP Composite

The PP sheet was made from PP pellets using a hot press machine at 170ºC with a pressure of 8 MPa for 5 min. In the manual rotation method, the CGFs were set lengthwise on the PP sheet. PP sheet layers (PL) stacked CGF layers (CGFL) were formed in steps similar to PL-CGFL-PL..., the outer layers were made by two layers of PP sheet. Composite samples were made using a hot press at 180ºC for 10 min at a pressure of 10 MPa and then cooled to another press with the help of two steel plates. The thickness of the resulting composites was kept at 2 mm. Figure 2 presents the processing method of PP/CGF composite and the temperature-time-pressure profile used for composite production.

Characterizations

Tensile features of the composite sample were measured using Shimadzu UTM (Model AG-1, Japan) with electrical weight cells of 6 kN physicomechanical properties following the ASTM-D 638-03 standard. The tensile test was achieved at a crosshead speed of 10 mm/min and a gauge length of 20 mm. According to ASTM-D 256 standards, the Izod impact strength was performed using an impact machine (model, Toyo Seiki Co., Japan). The dimensions of the sample were 63.5 × 12.7 × 3 mm^3. The hardness of the composite samples was tested by an HPE Durometer (model type 60578, Germany) according to DIN 53505 standard. The mechanical features of the unreinforced PP sheet were also tested according to the above-mentioned. All experiments were achieved at 25ºC ± 2ºC and relative humidity of 55 ± 5%. Reported values averaged over five measurements.

The SEM micrographs of untreated and treated composite samples were analyzed by a Zeiss, Evo 50 scanning electron microscope. The fracture edges of the specimens were embedded in an Al spit and covered with a thin layer of gold to disperse the electric charge throughout the test.

Water absorption of untreated and treated composite samples was determined by dipping the sample in a beaker covering water at 25ºC. After a constant time break, the sample was taken out of the water, drained, and weighed.

The composite sampling was conducted by the Accelerated Weathering Tester (Model Q-U-V, Q-Panel Company, USA). Untreated, SDS treatment and SDS pretreated benzoyl chloride treated composite samples were tested. The treatment differed between 60ºC ± 2ºC (sunshine) and 40ºC ±2 ºC (condensation) over 4 h of sunshine and 2 h of condensation for a period of 500 h. The specimens were dried in an oven for 0.5 h and measured in their tensile properties due to weather testing.

3. RESULTS AND DISCUSSIONS

Mechanical Properties of the Composites

The mechanical properties of the composites strongly influence the properties of the fibers and the matrix, the interfacial adhesion between the fibers and the matrix, the content of the fibers, and the length and orientation of the fibers. To determine the mechanical properties of composites, an interfacial bond between the fiber and the matrix must be observed.

Table 2: Formulations of untreated and treated PP/CGF composites

Ingredients (wt%)	Sample designation		
	PP/CGF	PP/SCGF	PP/SBCGF
PP	100	100	1000
CGF	0, 10, 20, 30, 40	–	–
SCGF	–	0, 10, 20, 30, 40	–
SDS pretreated benzoylated CGF	–	–	0, 10, 20, 30, 40

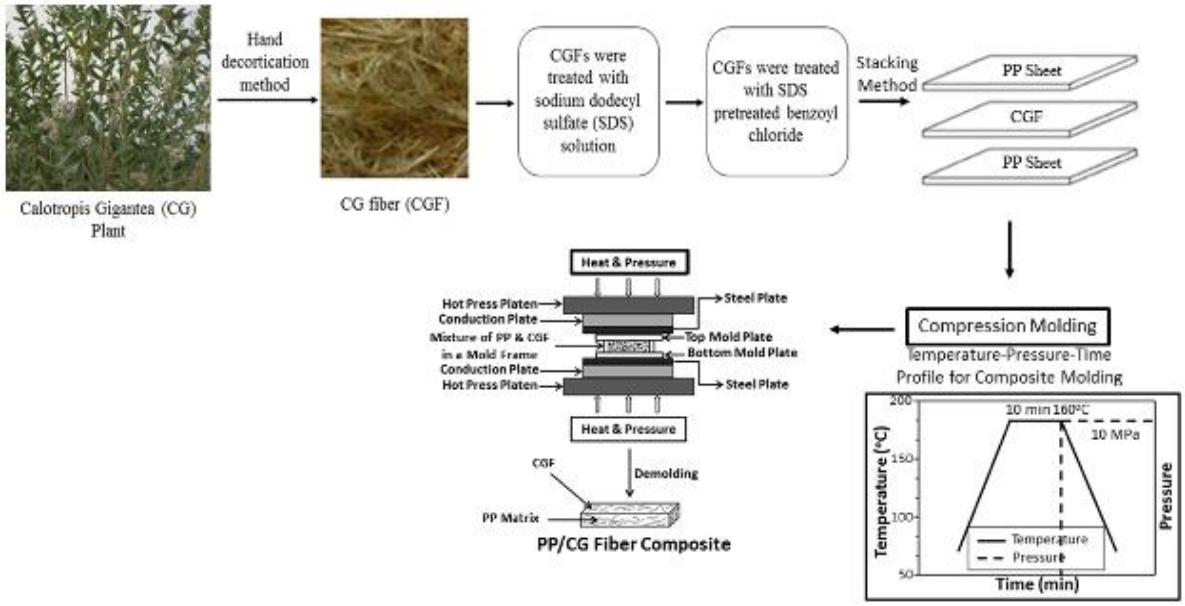

Figure 2 The schematic illustration of the manufactured PP/CGF composite and its processing profile

Tensile Features
Effect of CGF Loading

The tensile properties of untreated and interfacial modified composites based on fiber content are shown in Figures 3 and 4 (a). It can be observed that the composites reach the values of the tensile properties of the neat PP sample at 10 wt%. The fiber content (wt%) below the critical fiber content cannot improve the tensile properties of the composites. This may be due to the weak dispersion of fibers in the PP matrix resulting in improper interfacial adhesion between the fiber and the matrix. The test results indicate that the tensile strength and tensile modulus of PP are 28.2 MPa and 835.5 MPa, respectively. The inclusion of CGF in the PP matrix increases the tensile strength of the composite from 32.4 MPa to 38.7 MPa as the fiber content increases from 10 wt% to 30 wt%. According to other researchers [20, 21], tensile strength has improved with fiber content. This behavior is mainly responsible for the transfer of even stress from the matrix to the fiber. Anyway, the addition of more fiber content (40 wt%) did not increase the tensile strength. More than 30 wt% fiber content, have a tendency to decrease in tensile strength has been observed, probably due to an agglomeration effect on poorly dispersed fibers in the PP matrix at 40 wt% fiber content, resulting in a non-uniform transition to applied stress and weak fiber-matrix adhesion. In contrast, the inclusion of CGF in different fiber loading from 10 wt% to 30 wt% resulted in a continuous increase in the tensile modulus of the composites (Figure 4 (a)). As CGF's increased from 10 wt% to 30 wt%, tensile modulus increased to 18%, 31%, and 36% including 987.4 MPa, 1097.3 MPa, and 1135.2 MPa, respectively, compared to virgin PP. As the fiber content increases up to 30 wt%, the value of the tensile modulus increases and then decreases. Improvement of tensile modulus may be due to the reinforcing effect of fiber where stress is a transfer from the matrix to the fiber. The results indicate that untreated

fibers do not interact well with the PP matrix due to the presence of surface impurities. Composites containing 40% fiber loading show a decrease in tensile modulus. At higher fiber content (40 wt%), the agglomeration of fiber is found more, which leads to the deterioration of the mechanical properties of the composites. If PP was inadequately available, the fibers at higher fiber loading would no longer be surrounded by PP and voids would be created in the composite. It is known that most of the properties of composites were influenced by the presence of voids [22]. From the above results, composites with a fiber content of 30 wt% give maximum mechanical properties. Therefore, it is proven that 30 (wt%) fiber content is an optimum fiber content that can get a better combination of mechanical properties in PP/CGF composites.

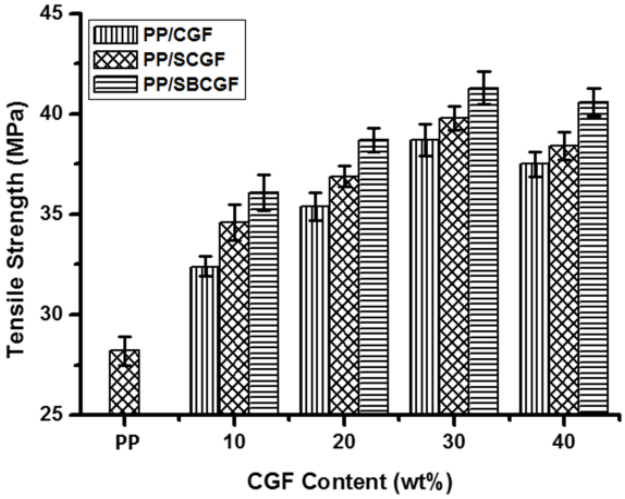

Figure 3. Tensile strength of PP/CGF, PP/SCGF, and PP/SBCGF composites at different fiber loading

Effect of SDS Treatment

Figure 3 shows the influence of SDS treatment on the tensile strength of PP/SCGF composite. With an increase in load level from 10 to 30 wt%, the tensile strength of PP/SCGF composite increased from 23%, 31%, and 41% to 34.6 MPa, 36.9 MPa, and 39.8 MPa, respectively, compared to virgin PP, then decreased with a further increase in CGF content (40 wt%). The surface of the CGF treated with SDS had long alkyl chains covalent bonding which increased the wettability with the help of PP matrix and it increased the interfacial bond between CGF and PP matrix. The presence of SDS in cellulose fibers is expected to reduce the hydrophilic character due to the formation of chemical interactions between the polar head group of SDS and the polar group of fibers [18]. One-way fiber orientation requires less fiber overlapping of the fibers and thus pulls out the fiber, reducing fiber agglomerations. Air penetration was less likely due to one-way fiber orientation. The entry of air creates a tip form which leads to weak stress transfer between the fiber and the matrix. After 30 wt% SCGF load, the tensile strength of composite reduces. This can be due to the loss of cellulose and the introduction of internal cracks in the fibers.

Figure 4(a) displays the correlation between tensile modulus and CGF load with and without SDS treatment. With an increase in load level from 10 to 30 wt%, the tensile modulus increased from 21% to 49% compared to virgin PP and then decreased with a further increase in CGF content (40 wt%). Currently available tensile modulus ranges from 1012.5 MPa to 1245.6 MPa using the same fiber content. This leads to friction in the interface region between the CGF particle and the PP matrix towards a rigid interface that impedes the movement of the polymer chain. Improvement of tensile modulus may be related to increased stiffness of composites due to CGF loading.

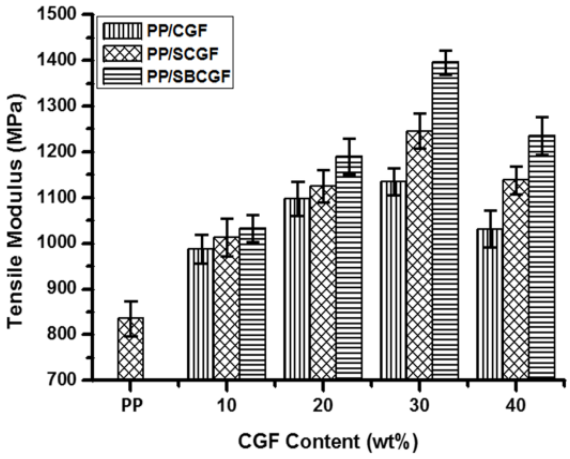

Figure 4 (a) Tensile modulus of PP/CGF, PP/SCGF, and PP/SBCGF composites at different fiber loading

Effect of Benzoylation Treatment
Benzoylating Treatment

The tensile properties of the PP/SBCGF composites are plotted in Figures 3 (a) and 4 (a). With an increase in load level from 10 to 30 wt%, the tensile strength and modulus of the PP/SBCGF composite increased from 28%, 37%, 47% and 24%, 42%, 67%, respectively, as

compared with virgin PP, then decreased with a further increase in CGF content (40 wt%). For chemical modification of fibers, improved tensile strength and modulus can be applied for better adhesion between fiber and matrix, and a small portion of hemicellulose is converted to benzoylated hemicellulose. Benzoylation similarly reduces polarity by wrapping the hydroxyl groups located in the cell wall of CGF. The hydrogen atoms of the –OH group have been replaced by the benzoyl group as illustrated in Figure 4b (iii).

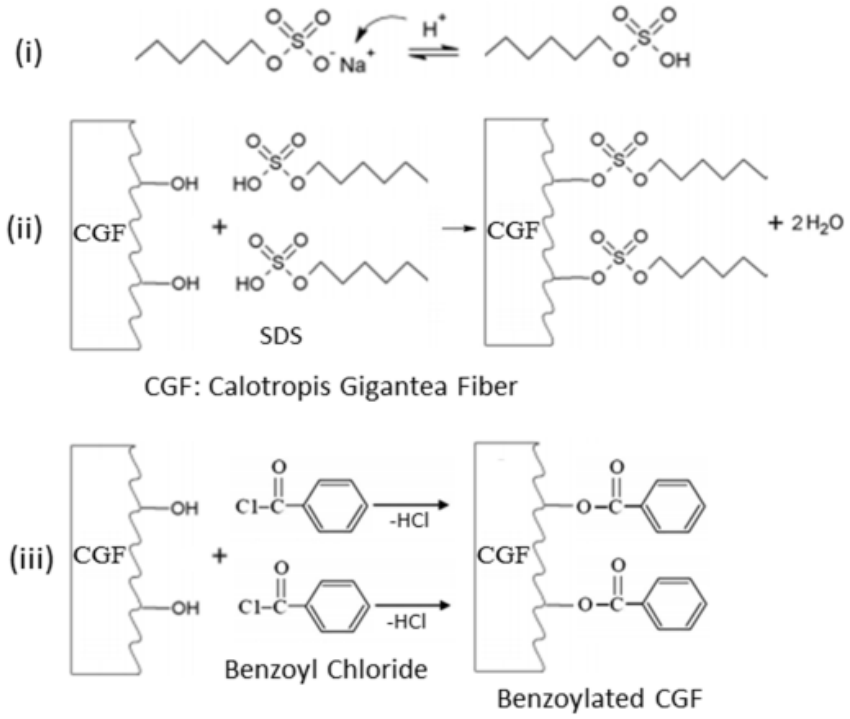

Figure 4 (b) (i & ii) Probable reaction mechanism of CGF with SDS, and (iii) reaction mechanism of CGF with benzoyl chloride

Figure 4 (b) (i & ii) Probable reaction mechanism of CGF with SDS, and (iii) reaction mechanism of CGF with benzoyl chloride Improving the mechanical properties of benzoylated composites, the introduction of the benzoyl group is applied to reduce the hydrophilicity of fibers, which gives the fibers a more hydrophobic character and therefore provides surfaces with better interaction of hydrophobic matrix [23]. SEM investigations of benzoylated fibers (Fig. 7, c) show that benzoylation makes the fiber surface very rough and increases fibrillation, and provides better mechanical interlocking with the PP. The improvement in tensile properties is due to the good bonding between benzoylated fibers and the PP matrix, which can be supported by the tensile properties of the composite from the SEM photomicrograph on the surface. The tensile properties of the SDS pretreated benzoylated fiber composites were found more than untreated fiber composites and more than SDS treated composites.

Impact Toughness and Hardness Features

Impact strength can be described as toughness or the ability of a hard material to withstand a sharp blow, such as from a hammer. The variation of impact strength with different CGF loading for untreated and treated (SDS or both SDS and benzoylation) CGF composites is shown in Figure 5. From the diagram, it was observed that the impact strength showed the same trend as the tensile features. The best properties were observed at 30 wt% CGF loading. This result suggests that the CGF was able to absorb energy due to the strong interfacial bond between the CGF and the PP. Over 30 wt% of CGF, impact strength decreases. More than 30 wt% of CGF was insufficient to wet the fiber with PP resin and there were many voids, resulting in a fiber-PP bond. For the treated PP/SCGF and PP/SBCGF composites, impact strength values have been observed to increase gradually with an increase in fiber content up to 30 wt%, indicating that the fiber

contributes positively to absorb impact energy. PP/SCGF and PP/SBCGF composites exhibited significantly higher impact strength values than untreated ones. SDS results in improved interfacial debonding of fiber with PP, perhaps owing to additional sites created for mechanical interlocking [24]. At this stage, the impact strength increased by 18% at 30 wt% fiber loading compared to untreated ones. On the contrary, the addition of benzoylated CGF to PP indicates an 18% improvement in impact strength compared to PP/SCGF composite. This enhancement in impact strength was due to the minimum interfacial debonding according to Mishra et al., [25]. The influence of fiber content on the hardness of PP/CGF, PP/SCGF, and PP/SBCGF composites is illustrated in Figure 6. In general, fibers that increase the modulus of composites increase their hardness in thermal applications. It was found that the hardness of the two types of modified composites increased by 3, and 6%, respectively, compared to the PP/CGF composite, at 30 wt% CGF content. The inclusion of fillers in the PP matrix reduces polymer chain mobility in rigid composites. Better hardness is seen in a composite of two types of treatment than in untreated composites. This can be attributed to the good dispersion of CGF in PP so that the voids are reduced and there is a strong interfacial bond between PP and CGF.

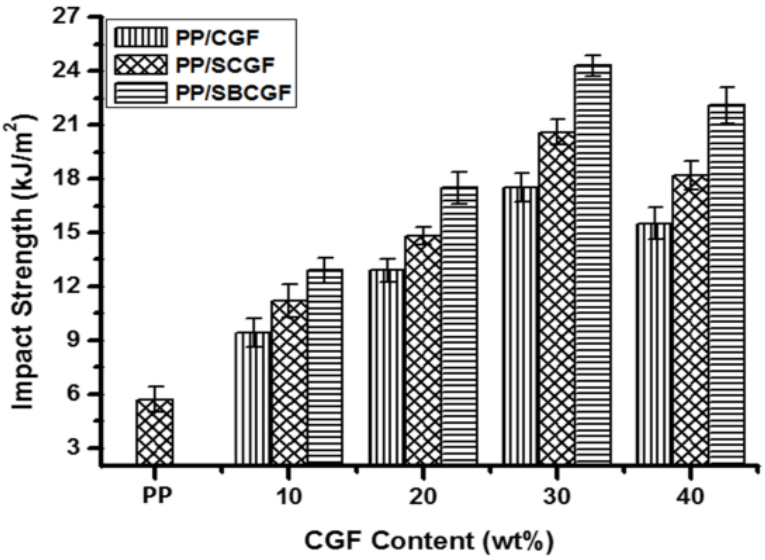

Figure 5 Impact strength of PP/CGF, PP/SCGF and PP/SBCGF composites at the different fiber content

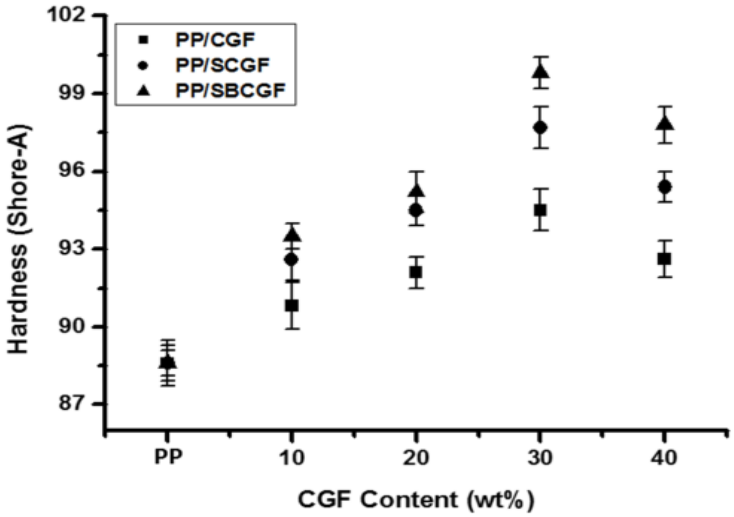

Figure 6 Hardness of PP/CGF, PP/SCGF, and PP/SBCGF composites at the different fiber content

Morphological Study

For CGF-PP adherence, which was inferred from the mechanical properties predicted in the previous section, tensile specimens of SEM were used to identify fracture surfaces. Figure 7 displays SEM images of fracture surfaces of (a) PP/CGF, (b) PP/SCGF, and (c) PP/SBCGF composites. The SEM micrographs (Figure 7, a) show weak adhesion between the CGF surface and the matrix. Many large voids were enclosed in the CGF and the fibers lacked contact with the matrix due to the incompatibility of these elements. It has been observed that the interfacial structure of this composite cannot effectively stress transfer. This observation was consistent with the low tensile strength values described in Figure 3. Treated specimens showed better bonding than the untreated ones. SDS treated composites to improve the dimensional stability and surface roughness of CGF and thus improve bonding with PP (Fig. 7, b). We further noticed that the CGF was partially wetted by the PP. For PP/SBCGF composites (Fig. 7, c), the CGF's were somewhat elongated during the fracture process and most of the PP matrix was attached to the surface of the CGF's and adhered. Fiber seems to be less prolonged for PP/SCGF composites than PP/CGF composites. This has confirmed a good interaction between CGF and PP. SEM photomicrographs show evidence of increased mechanical properties and moisture absorption properties of PP/CGF composites by SDS treatment or both SDS and benzoylation treatment.

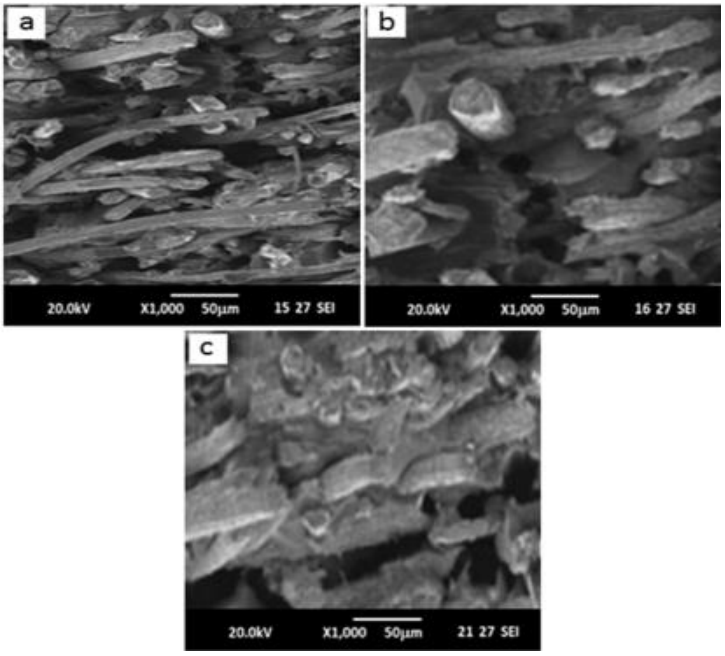

Figure 7 SEM images of the fracture surfaces of (a) PP/CGF (untreated), (b) PP/SCGF, and (c) PP/SBCGF composite at 30 wt% fiber content

Water Absorption (WA) Behavior

The results of WA values for PP/CGF, PP/SCGF, and PP/SBCGF composites as a function of water soaking time (24 h) against CGF content are presented in Figure 8. Soaking time and the amount of CGF content are substantial issues, which affect the WA of the composite. The WA of PP was 0.04% which was negligible than 40 wt% CGF-reinforced PP composite (1.53%). From the test results, we can see that the amount of WA was obtained after 24 hours immersion in water in the range of 0.58% for 10 wt% fiber loading to 1.53% for 40 wt% fiber loading due to the higher availability of cellulose –OH groups that can absorb water. Due to the presence of micro-voids in the CGF-PP interface, natural fiber-reinforced composites typically exhibit transient moisture absorption due to incomplete wetting of CGF through PP [26]. At a loading level of 40 wt%, the WA of PP/SCGF composite (approximately 60%) decreases compared to the untreated composite. This may be due to the interfacial bond between the PP matrix and the CGF. In contrast, benzoylated composites (PP/SBCGF) in 40% fiber content showed about 65% WA less than in untreated samples. It was further

found that the WA of PP/SBCGF composite was less than that of PP/SCGF composite in 40% fiber content. Benzoylation of CGF leads to a decrease in the hydrophilicity of the fiber and roughs the surface of the fiber. The reduction of the hydrophilic nature makes the fiber more compatible with hydrophobic PP and improves the adhesion between rough surface CGF and PP. This reduction in the hydrophilic nature of CGF and better interfacial adherence between CGF and PP is the cause of WA reduction by benzoylated CGF composites.

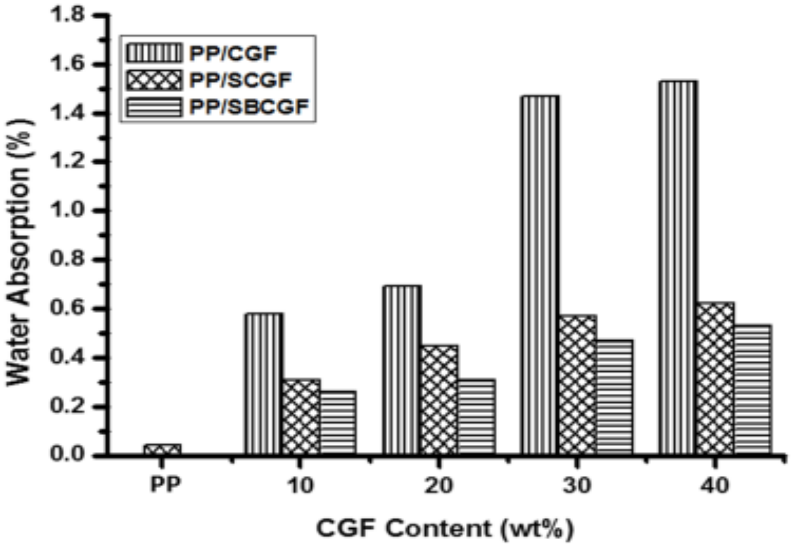

Figure 8 WA values of PP/CGF, PP/SCGF, and PP/SBCGF composites against different fiber loading as a function of water soaking time (24 h)

Weather Effects

Untreated and treated specimens were exposed to simulated sunshine and severe weather tests over 500 h of condensation in the periodic cycle. The tensile properties such as tensile strength (TS) and tensile modulus (TM) of the specimens were measured periodically. Figures 9 and 10 represent a reduction in TS and TM of the specimens due to weather. In most observations, the loss of the TS of the untreated specimen was about 23%, while the treatment composite of PP/SCGF and PP/SBCGF was about 11.4% and 6.7%, respectively. Similarly, the TM loss of untreated specimens was about 21%, whereas PP/SCGF and PP/SBCGF specimens were about 10% and 5%, respectively. Weather tests showed that untreated specimens lost tensile features (TS and TM), but treated specimens retained tensile features for decades despite being exposed to intense weather for 500 h. The PP/SCGF specimen exhibits greater weather acceptance and is more durable than other specimens.

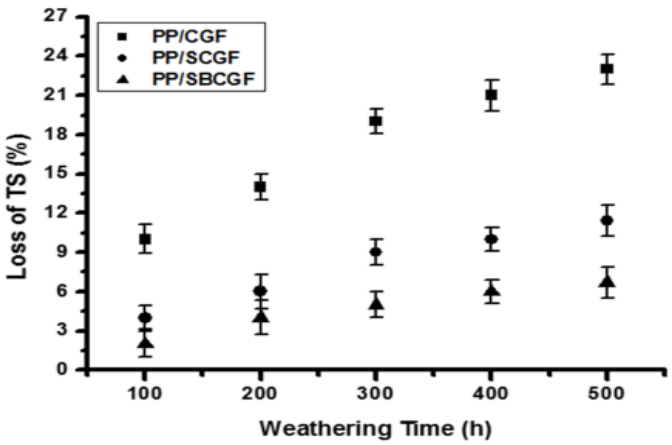

Figure 9 Loss of tensile strength of the untreated and treated composites due to weather simulation

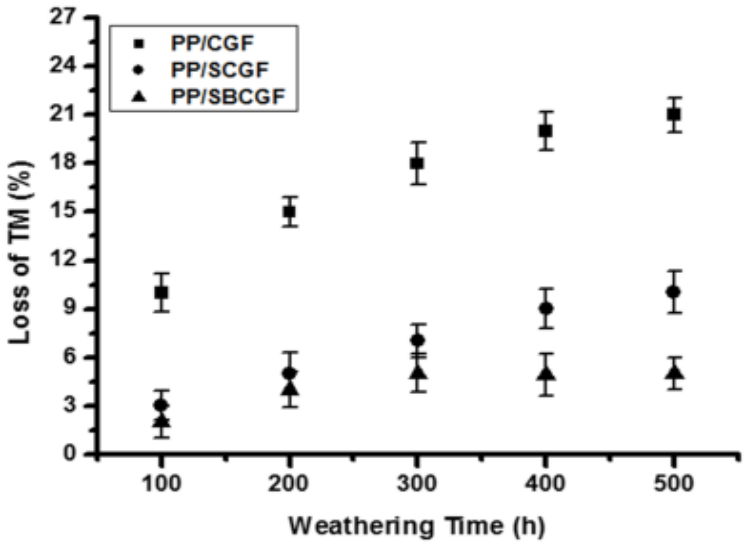

Figure 10 Loss of tensile modulus of the untreated and treated composite due to weather simulation

4. CONCLUSIONS

In this experiment, the effect of SDS and benzoylation treatment of CGFs on the basic properties of PP/CGF composites was investigated and analyzed. From the above, we can conclude the following:

(1) Mechanical properties such as the tensile strength, tensile modulus, impact strength, and hardness of the PP/CGF composites were increased to the optimal fiber content of 30 wt%. However, 40% fiber load composites have lower tensile properties than 30% fiber load composites.
(2) The mechanical properties of PP/SCGF and PP/SBCGF composites were higher than those of untreated composites.
(3) SEM investigation of the fractured surfaces of the treatment composite revealed a rich bond between PP and CGF's.
(4) The WA behavior of the treated specimens showed a remarkably lower tendency than that of the untreated specimens. Weather studies have shown that the loss of TS and TM in treated specimens may be less than that of untreated specimens in terms of their decay time.

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